

Templating Effects on the Enhanced Formation of C54-TiSi

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Abstract

Titanium disilicide (C54-TiSi₂) is a low resistivity silicide (15-20 $\mu\Omega$ -cm) suitable for applications as contacts and interconnects between devices in Metal Oxide Semiconductor Field Effect Transistor (MOSFET) semiconductor technology. C54-TiSi₂ is the most stable silicide in the Ti-Si system. It forms in a device manufacturing process when a Ti/Si bilayer is subjected to rapid thermal annealing (RTA) in N₂. During this process, a metastable and high resistivity (60-70 $\mu\Omega$ -cm) disilicide (C49-TiSi₂) forms first. The desired disilicide (C54-TiSi₂) is obtained on further heating. Low C49-to-C54 transformation temperatures are desirable to assure suitable electrical properties as well as to minimize dopant diffusion and thin-film agglomeration effects. This research studies the mechanisms for enhanced C54 formation at lower temperatures during rapid thermal annealing. A variety of thin-film specimens in the Ti-Si, Ti-Ta-Si, and Ti-Mo-Si systems were prepared by sputtering. These were subjected to various RTA procedures and quenched to room temperature. Cross-sectional TEM samples were prepared using the tripod-polishing method. Convergent beam electron diffraction (CBED), conventional bright-field imaging, high-resolution (HRTEM) imaging in conjunction with Fast Fourier Transform analysis of lattice images, and computer crystal simulations were used extensively to study the phase evolution of these materials. A new method for enhancing C54 formation was suggested after systematic microstructural observations and phase identification for the ternary alloy cases (Ti-Ta-Si) and (Ti-Mo-Si) after RTA in N₂. Recent work reported in the literature on isothermal annealing of Ti-Ta/Si and Ti/Mo/Si specimens, attributes the enhanced C54 formation to a template mechanism due to the hexagonal C40-TaSi₂, and C40-(Ti_xMo_{1-x})Si₂. In the present research, however, the enhancement is related to the presence of the hexagonal Ti₅Si₃ or its isomorphous compounds (Ta₅Si₃, (Ti_xTa_{1-x})₅Si₃) which catalyze nucleation of C54-TiSi₂. There is a pseudomorphic relation between (040) C54-TiSi₂ and (300) Ti₅Si₃, which has a structural mismatch of 6-7% but otherwise good correspondence between atomic positions and atom types. This relation suggests that underlayers of Ti₅Si₃ would promote C54 formation at lower annealing temperatures. First experiments using underlayers of crystalline Ti₅Si₃/Si on which amorphous TiSi_{1.3} was deposited found a drop in the C54 formation temperature in support of this proposed enhancement mechanism.

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Mouroux, A.: The reactive formation of TiSi₂ in the presence of refractory metals (from V to W), Royal Institute of Technology, Department of Electronics, Solid State Electronics, Stockholm, Sweden 1997.

ABSTRACT

Titanium disilicide (TiSi₂) has been the favoured material for contact metallisation in recent Si devices. The formation of TiSi₂ usually begins with the high resistivity C49 phase as a result of the Ti-Si interaction at about 300-550 °C and finishes with the low resistivity C54 phase through the C49-C54 phase transformation at about 700 °C. The C49-C54 transformation becomes increasingly difficult as the device dimensions are scaled down and remains a technological challenge for dimensions below 0.5 µm. In this thesis, a thin layer of Mo deposited between Ti film and Si substrate is used to promote the formation of the C54 phase at temperature 100 °C lower than for the C49-C54 transformation. On narrow Si lines down to 0.25 µm width, the influence of Mo on the formation of TiSi₂ is even more pronounced than on blanket substrates; lower sheet resistance with smaller scatter is obtained in the presence of Mo than without. The enhancement in the formation of the C54 phase is interpreted as a consequence of epitaxial effects where the formation of C40 (Mo,Ti)Si₂ plays a key role. The validity of the template mechanism is verified by replacing Mo with Ta and Nb. The idea of using Ta and Nb comes from the fact that TaSi₂ and NbSi₂ have the same crystallographic structure and comparable lattice parameters as (Mo,Ti)Si₂. The epitaxial mechanism is confirmed by lattice imaging with a high-resolution microscope. In order to gain an insight into the thermodynamics of the ternary systems, the pseudo binary phase diagram of TiSi₂-NbSi₂ is studied. Three phase domains are identified, i.e. 1) C54 (Ti,Nb)Si₂ with Nb varying from 0 to 10 % at the metal sites, 2) a mixture of C54 and C40 (Ti,Nb)Si₂ with Nb being 10 to 25 % at the metal sites, and 3) C40 (Ti,Nb)Si₂ with Nb varying from 25 to 100 % at the metal sites. The resistivity of (Ti,Nb)Si₂ C54 increases by 1.2 µΩ cm per at. % Nb when the Nb concentration varies from 0 to 10 % at the metal sites. The presence of the refractory metals (Ta, Nb or Mo) at the Si/Ti interface modifies the energetic factors for the formation of C54 TiSi₂. The formation of C49 TiSi₂ is hindered and that of C54 is enhanced. With a Mo or Nb interposed layer, the phase of C54 TiSi₂ can be obtained at temperatures as low as 450 °C. Moreover, if a continuous silicide layer in the C40 structure is formed at the Si/TiSi₂ interface, it is the Si diffusion through this interfaced layer that is the controlling factor for the C54 TiSi₂ growth. Furthermore, the use of an interposed layer of Mo, Ta or Nb generally improves the surface morphology and morphological stability: the TiSi₂ formed has a smoother surface and interface and is more resistant to agglomeration.

Key words: Titanium disilicide TiSi₂, contact metallisation, phase formation, refractory metals, sub-micron technology, template growth, very large scale integration, VLSI, interconnection.

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**Understanding the formation of TiSi_2 in the presence of refractory metals for Si
technology
PhD.Thesis: Aliette Mouroux**

ABSTRACT

Titanium disilicide (TiSi_2) has been the favoured material for contact metallisation in recent Si devices. The formation of TiSi_2 usually begins with the high resistivity C49 phase as a result of the Ti-Si interaction at about 300-550 °C and finishes with the low resistivity C54 phase through the C49-C54 phase transformation at about 700 °C. The C49-C54 transformation becomes increasingly difficult as the device dimensions are scaled down and remains a technological challenge for dimensions below 0.5 μm . In this thesis, a thin layer of Mo deposited between Ti film and Si substrate is used to promote the formation of the C54 phase at temperature 100 °C lower than for the C49-C54 transformation. On narrow Si lines down to 0.25 μm width, the influence of Mo on the formation of TiSi_2 is even more pronounced than on blanket substrates; lower sheet resistance with smaller scatter is obtained in the presence of Mo than without. The enhancement in the formation of the C54 phase is interpreted as a consequence of epitaxial effects where the formation of C40 (Mo,Ti) Si_2 plays a key role. The validity of the template mechanism is verified by replacing Mo with Ta and Nb. The idea of using Ta and Nb comes from the fact that TaSi_2 and NbSi_2 have the same crystallographic structure and comparable lattice parameters as (Mo,Ti) Si_2 . The epitaxial mechanism is confirmed by lattice imaging with a high-resolution microscope. In order to gain an insight into the thermodynamics of the ternary systems, the pseudo binary phase diagram of TiSi_2 - NbSi_2 is studied. Three phase domains are identified, i.e. 1) C54 (Ti,Nb) Si_2 with Nb varying from 0 to 10 % at the metal sites, 2) a mixture of C54 and C40 (Ti,Nb) Si_2 with Nb being 10 to 25 % at the metal sites, and 3) C40 (Ti,Nb) Si_2 with Nb varying from 25 to 100 % at the metal sites. The resistivity of (Ti,Nb) Si_2 C54 increases by 1.2 mW cm per at. % Nb when the Nb concentration varies from 0 to 10 % at the metal sites. The presence of the refractory metals (Ta, Nb or Mo) at the Si/Ti interface modifies the energetic factors for the formation of C54 TiSi_2 . The formation of C49 TiSi_2 is hindered and that of C54 is enhanced. With a Mo or Nb interposed layer, the phase of C54 TiSi_2 can be obtained at temperatures as low as 450 °C. Moreover, if a continuous silicide layer in the C40 structure is formed at the Si/ TiSi_2 interface, it is the Si diffusion through this interfaced layer that is the controlling factor for the C54 TiSi_2 growth. Furthermore, the use of an interposed layer of Mo, Ta or Nb generally improves the surface morphology and morphological stability: the TiSi_2 formed has a smoother surface and interface and is more resistant to agglomeration.

Key words: *Titanium disilicide TiSi_2 , contact metallisation, phase formation, refractory metals, sub-micron technology, template growth, very large scale integration, VLSI, interconnection.*

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Reduction of the Transition Temperature of C54TiSi

2 through a Ta Interlayer

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We examined the phase transition of TiSi₂ from C49 to C54 by introducing a thin Ta interlayer between Ti film and Si(111) substrate. The Ta interlayered samples were prepared by depositing 5 Å Ta and 100 Å Ti films sequentially on Si(111) substrates in the UHV system. As control samples,

100 Å Ti films were deposited on Si(111) substrates. The deposited substrates were annealed insitu at temperatures between 500,750 fñC with 50 fñC increments for 10 min. The TiSi₂ formed in UHV system was analyzed by using XRD, AES, SEM and a four-point probe. The control samples exhibited the transition temperature of 750 fñC from C49 to C54. However, the TiSi₂ samples with 5 Å of Ta interlayer showed significant reduction of the phase transition temperature of C54 TiSi₂. The XRD pattern analysis indicated that the transition temperature of TiSi₂ from the C49 to C54 phase was lowered by about 200 fñC with the addition of 5 Å Ta interlayer. The sheet resistance measurement showed lower resistivity of Ta interlayered samples of TiSi₂ due to the reduction in the formation temperature of C54 phase. The SEM micrographs showed that the TiSi₂ with 5 Å Ta interlayer suppressed the surface agglomeration, significantly. The AES analysis data indicated that the **titanium** silicide formed at high temperature (>500 fñC) showed a 1:2 stoichiometry of Ti:Si and small amounts of impurities in the silicide film and the interface between silicide and Si substrate.

1. INTRODUCTION **Titanium** silicide has been the most widely used and studied silicide among refractory metal silicides because of its great importance in the technology and science of Si-based semiconductor devices. The low resistivity and high thermal stability of TiSi₂ are strong points to apply this material to current device process. However, the surface of **titanium** silicide becomes significantly rough when it forms C54 TiSi

2 at high temperatures (>650 fñC). TiSi₂ structure exhibits polymorphic structures which are metastable C49 phase (resistivity:

60,70 u\Omega-cm, base-centered orthorhombic) and a stable C54 phase (resistivity: 15.20

u)Omega -cm, face-centered orthorhombic) [1,2]. The achievement of the low resistivity phase of TiSi₂ requires high temperature anneals(

>650 °C) to transform the C49 phase to C54 phase [3]. The transition temperature of TiSi

2 from C49 to C54 has been reported to depend on the Ti film thickness,

linewidth, dopant concentration and the Si substrate orientations [4-6]. These days the scaling down of device dimensions in the very-large-scale-integration technology causes the phase formation of C54 TiSi

2 phase to become more difficult without causing agglomeration [7,8].

There were several researches reported on the reduction of the transition temperature of TiSi

2, such as a

refractory metal implantation and a preamorphization of Si substrate [9-11]. The mechanism responsible for the lowering the transition temperature of C54 TiSi₂ has been explained as the increase in the nucleation density of the C54 phase in the C49 matrix and the reduction in the energy barrier to nucleate the C54 phase. These processes lowered the transition temperature of TiSi₂ to some extent. Another approach that is the deposition of a thin layer of Ta between Ti film and Si substrate as an interlayer has been tried [12]. The deposition of the thin layer of refractory metal of Ta is expected to affect the transition temperature of C54 TiSi

2. The mechanism responsible for the reduction of C54 phase formation by

introducing a thin Ta layer is considered due to a crystallographic orientation matches between two metal phases. The TaSi₂ phase which is the C40 phase (hexagonal) has the basal plane which exhibits similar atomic arrays with the (001) planes of C54 TiSi₂. This crystallographic similarity may enhance the formation of C54 TiSi

2 and reduce the transition temperature.

In this experiment, we deposited a 5 nm of Ta layer as an interlayer between 100 nm Ti film and Si(111) substrate and then annealed in the ultrahigh vacuum system at the temperatures between 500 to 750 °C with 50 °C increments for 10 min. We examined two sets of TiSi₂ formation, with and without 5 nm of Ta interlayer to investigate

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Fig. 1. XRD patterns of Ti-silicide without (a) and with (b) Ta interlayer as a function of annealing temperatures.

to investigate the effect of this Ta interlayer on the phase transition temperature. The physical and electrical properties of TiSi₂ films were examined by using SEM, AES, XRD and a four-point probe. With these results, we will explain the reduction of phase transition temperature of TiSi

2 based on the crystallographic orientation and will correlate the surface roughness with the electrical property.

II. EXPERIMENT The substrates used in this study were Si(111)-oriented substrates with resistivities of 0.68, 0.72 Ω cm (n type, P doped). The wafers were cleaned by UV/ozone exposure and spin etching with HF + H

2O + ethanol, 1:1:10 at room temperature before loading into the ultrahigh vacuum (UHV) chamber [13-15]. The system equipped with a turbopumped-loading chamber and cryopumped main chamber was used to deposit and anneal the Ta or Ti layers on the Si substrates. After loading the wafers, the heat cleaning of Si substrate was carried out by heating

Fig. 2. SEM micrographs of TiSi₂ formed from 100 Å Ti deposition on Si(111) substrates and in-situ annealed at (a) 700 °C and (b) 750 °C for 10 min.

up to a temperature of 800 °C for 10 min to desorb the residual contaminants and hydrogen. Two sets of samples were prepared in a multi source-electron beam UHV chamber: One set of samples were prepared with deposition of 100 Å Ti on Si(111) substrates as control samples. Another set of samples were made by introducing 5 Å Ta layer between 100 Å Ti and Si(111) substrates. The base pressure in the UHV chamber was 1.2×10^{-10}

10 Torr and

the Ta and Ti films were deposited consecutively without breaking the vacuum. After metal depositions at

room temperature, the substrates were in-situ annealed to the temperatures of the range of 500, 750 °C with 50 °C increments for 10 min. After annealing the samples, the physical and electrical properties are examined by a four-point probe, XRD, SEM and AES.

III. RESULTS AND DISCUSSIONS The XRD was used to analyze the phase formation of TiSi

2 as summarized in Fig. 1. The C49 phase of TiSi₂ was found on the control samples annealed between 500

and 700 °C, as shown in Fig. 1(a). When annealed

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Fig. 3. SEM micrographs of TiSi₂ formed from 100 Å Ti and 5 Å Ta deposition on Si(111) substrates and in-situ annealed at (a) 700 °C and (b) 750 °C for 10 min.

at 750 °C, the C49 phase was completely transformed into the C54 phase on the control sample. The peaks related with the C54 phase was identified at 750 °C anneal. The transition temperature of 100 Å thick Ti film was to some extent high in agreement with the thickness dependence in the case of the Ti films less than 200 Å thick. And the highest intensity peak of C54 TiSi₂ phase formed at 750 °C was to be the (004) plane. Figure 1(b) shows the XRD patterns from the samples of

100 Å Ti/5 Å Ta on the Si(111) substrates. The (131) peak of C49 TiSi

2 was formed at the sample when annealed at 500 °C. However at 550 °C, peaks related with

C49 TiSi₂ phase were not observed and the (202) peak of C54 TiSi

2 was shown clearly at $2\theta = 30.1^\circ$. The intensity of (202) peak of C54 TiSi

2 was increased with the anneal temperatures. The C54 (004) peak of TiSi

2 was found at temperatures greater than 700 °C, but the main peak

was still the (202) peak of C54 TiSi₂. The results of XRD indicated that the 5 Å Ta interlayer between 100 Å Ti and Si(111) substrate lowered the formation temperature of C54 TiSi₂ by about 200 °C and changed the main diffraction peak of C54 TiSi

2 from (004) plane to

Fig. 4. The change in sheet resistance of Ti-silicide without and with Ta interlayer on Si(111) substrate as a function of annealing temperatures.

(202) plane, suggesting a crystallographic feature variation. Such a variation of main the peak of C54 TiSi

2 is considered due to the change in the crystallographic

orientation by adding Ta interlayer. Figure 2 shows the changes in the surface morphologies of the TiSi₂. The control samples without Ta interlayer started to show surface agglomeration already at 700 °C (Fig. 2(a)) and accelerated to above 750 °C (Fig. 2(b)). But the samples with Ta interlayer, as shown in Fig. 3, exhibited no agglomeration and relatively smooth surfaces at high temperatures. This SEM examination indicated that the 5 Å Ta interlayer suppressed the surface agglomeration of C54 TiSi

2 phase at high temperatures. Figure 4 shows the change in sheet resistance of the Ti-silicide layers formed with and without being deposited on the Si(111) substrates as a function of annealing temperatures. The resistance of control samples of TiSi₂ varied significantly depending on the annealing temperatures. The increased resistance above 700 °C is due to the surface agglomeration of the C54 phase, as shown in Fig. 2. However, the C54 phase formed on the samples with the Ta interlayer showed relatively low resistance even at high temperatures due to the suppression of surface agglomeration in accordance with the SEM micrographs, as shown in Fig. 3. Therefore, we could conclude that as a result of the 5 Å Ta layer addition between the 100 Å Ti layer and Si substrate, the electrical and physical properties of the Ti-silicide were improved significantly. AES depth profiles of the silicide formed at the temperature of 500 °C are shown in Fig. 5. Both of the samples formed at 500 °C showed Ti-silicide formation with an atomic ratio of approximately Ti:Si = 1:2 at the anneal temperature irrespective of whether or not the Ta interlayer was present. Therefore the reaction between Ti and Si was started even at 500 °C heat treatment

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Fig. 5. AES depth profiles of (a) 100 Å Ti, and (b) 100 Å Ti/5 Å Ta deposited and annealed at 500 °C for 10 min.

and there was no Ti layer left which did not react with Si. During the silicide formation in the Ti-Si and Ta-Si systems, Si was reported to be the dominant diffusing element [16]. And in this Ti-Ta-Si system AES data showed Ta interlayer remained between Ti and Si substrate, as shown in Fig. 5(b). Therefore,

we can consider the Ta interlayer remains at the interface during the titanium silicide formation and enhances the formation of C54 TiSi₂. From the above results, the 5 Å Ta interlayer introduction into the 100 Å Ti/Si(111) system can reduce the transition temperature of C54 TiSi₂

by about 200 °C and suppress the surface agglomeration of C54 phase

significantly. We already reported that the transition temperature of TiSi₂

deposited by Ti single source less than 200 Å increased with the decrease in Ti film. In this

experiment, we found that the transition temperature of TiSi₂

was reduced by the addition of 5 Å Ta interlayer. Influence of Ta interlayer was very positive but the way

the Ta interlayer changed the formation mechanism of C54 TiSi₂

could not be understood clearly. We can explain such an enhanced formation of C54 TiSi₂

phase by considering the crystallographic orientation. The (001)

planes of TaSi₂, which have the C40 structure, are very closely lattice-matched to the (001) plane of C54 TiSi₂

2. As shown in Fig. 6, the Si atomic array of both C40

and C54 phases exhibit the similar hexagonal array and the inter-atomic closed distances are similar. The atomic distances of Ti-Si and Si-Si in the C54 TiSi₂ phase are 2.74 Å and 2.75 Å respectively. And those of Ta-Si and

Fig. 6. The structures of (a) C54 TiSi₂ phase and (b) C40 TaSi₂ phase.

Si-Si in the C40 TaSi₂ phase are 2.76 Å and 2.76 Å respectively. Therefore it may promote the nucleation and growth of C54 TiSi₂. This is a crystallographic explanation of this significant reduction of C54 phase formation temperature.

IV. SUMMARY We have studied the reduction of transition temperature of C54 TiSi₂

phase through a 5 Å Ta interlayer deposition between the 100 Å Ti film and Si(111) substrate. Apparently the Ta interlayer affected the titanium silicide formation process, significantly. The C54 TiSi₂ transition temperature was lowered by about 200 °C and the agglomeration of the C54 phase formed at high temperatures (>700 °C) was suppressed. With the addition of the Ta interlayer, the formed **titanium disilicide** showed excellent electrical and physical characteristics.

The role of the Ta interlayer is assumed to promote the growth of C54 phase of TiSi₂

and suppress the surface agglomeration of TiSi₂. The C54 TiSi₂ phase has a hexagonal atom array in (001) plane and the C40 TaSi₂

has also the hexagonal array in the basal plane similar to

the (001) plane of C54 phase. Our results suggest that the mechanisms responsible for the suppression of surface agglomeration and reduction of the transition temperature should be related with this similarity of C40 and C54 structures.

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